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Flow-tube reactor experiments on the high temperature oxidation of carbon weaves

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Overview

Under entry conditions carbon weaves used in thermal protection systems (TPS) decompose via oxidation. Modeling this phenomenon is challenging due to the different regimes encountered along a flight trajectory. Approaches using equilibrium chemistry may lead to over-estimated mass loss and recession at certain conditions. Concurrently, there is a shortcoming of experimental data on carbon weaves to enable development of improved models. In this work, a flow-tube test facility was used to measure the oxidation of carbon weaves at temperatures up to 1500 K. The material tested was the 3D carbon weave used for the heat shield of the NASA Adaptive Deployable Entry and Placement Technology, ADEPT [1]. Oxidation was characterized by quantifying decomposition gases (CO and CO_2), by mass measurements, and by microscale surface analysis. The current set of measurements contributes to the development of finite rate chemistry models for carbon fabrics used in woven TPS materials.

Experiment

Experiments were performed at the flow tube reactor facility at SRI [2]. The facility consists of a furnace-heated, vacuum-pumped quartz tube, equipped with a series of mass flow meters, pressure transducers, and temperature controllers (Figure 1). Inert gases, used during heating and cooling, and oxidizers (O_2 and CO_2 in the present work) are flown at controlled rates, while measuring the environmental pressures. Material decomposition products are quantified using a residual gas analyzer mass spectrometer which is calibrated in-situ using neat gases. A dedicated sample holder, shown in Figure 2, was developed to accommodate a flexible 3D carbon weave, flush-mounted with the wall.

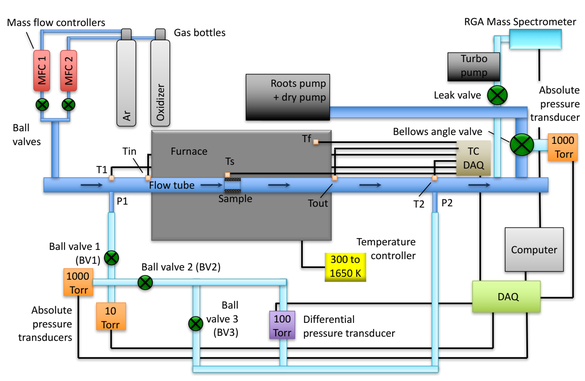

Figure 1. Schematic of the experimental setup.

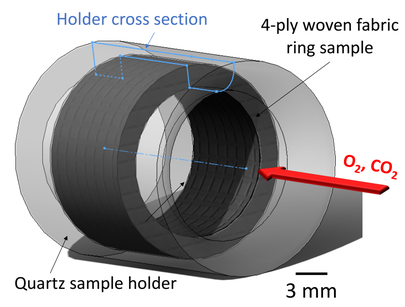

Figure 2. Schematic of the sample assembly.

Materials characterization

Scanning electron microscopy (SEM) images of the samples prior to and after oxidation revealed:

- smooth surface for the PAN-based fibers of the virgin material;
- oxidization by pitting of the fibers' surface, similar to the oxidation of rayon-based fibers observed in previous investigations [3];
- increased pitting with temperature, leading to fibers thinning and loss of tow compactness;
- incremental fraying of the woven structure that indicates ease for the shear flow to remove material by mechanical erosion [4].

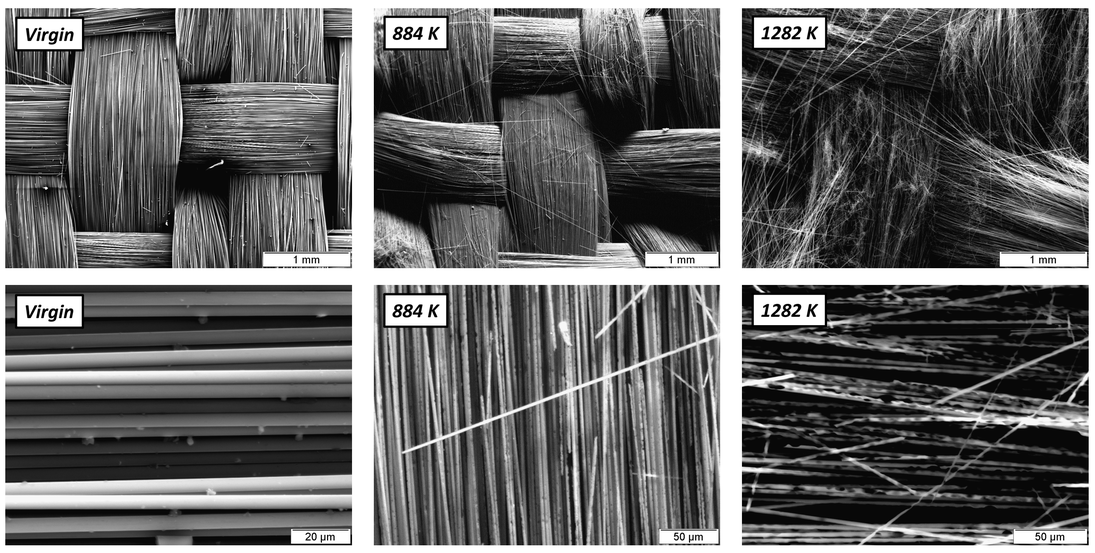

Figure 5. Scanning electron micrographs of test samples at different oxidation temperaturea. Top row: low magnification. Bottom row: high magnification.

Results

Experiments were carried out at several temperatures in O_2 and CO_2 for approximately 10 minutes oxidation time. O_2 tests yielded increasing mass loss up to 60% for the highest temperature. Negligible loss was measured in CO_2.

Table 1. Summary of experimental measurements

Sample	Gas	$\dot{m}$, mg/s	T, K	t, s	P_1, Pa	ΔP, Pa	m, g	$\Delta m/m$, %
1	O_2	2.34	1475	610	127	80	0.785	61.0
2	O_2	2.36	1282	608	122	36	0.869	37.6
3	O_2	2.34	1083	604	110	28	0.784	14.0
4	O_2	2.36	884	645	102	20	0.893	2.0
5	O_2	2.36	785	610	101	17	0.798	1.2
6	O_2	2.34	984	601	114	26	0.870	6.3
7	O_2	3.34	1379	607	124	60	0.841	60.8
9	O_2	2.34	881	609	100	27	0.921	4.2
11	CO_2	2.27	1476	608	94	33	0.909	2.6
12	CO_2	2.27	1281	600	84	n.a.	0.813	1.6
13	CO_2	2.27	1086	601	82	n.a.	0.819	1.7
14	CO_2	2.27	1378	601	93	n.a.	0.685	1.8
15	CO_2	2.27	1475	1199	96	n.a.	0.769	2.8

Mass spectrometry measurement of gaseous decomposition products during oxidation revealed predominant CO_2 production at low temperatures. At temperatures above 1100 K, CO prevailed instead. No O_2 consumption was observed at temperatures below 800 K.

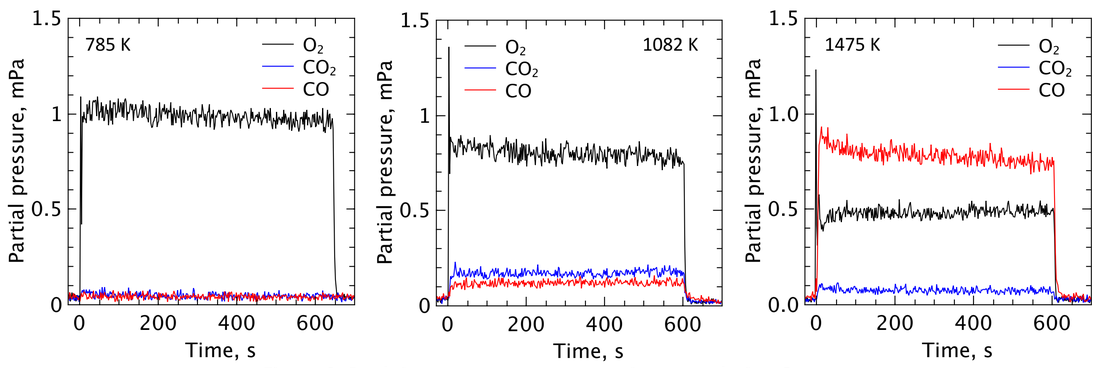

Figure 3. Real time mass spectrometry signature during O_2 tests.

Quantification of oxidation products confirmed CO_2 production at low temperatures and increasing CO production at increasing temperature. Total computed carbon loss from the gaseous products underestimated the total measured mass loss at high temperatures. This observation suggested a strong influence of material removal by flow shear (spallation) at high temperatures. Visual observations of the sample during oxidation revealed indeed a significant mechanical material removal.

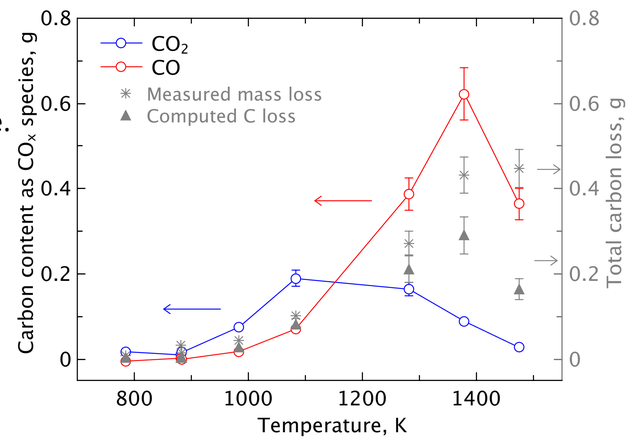

Figure 4. Quantification of mass loss and oxidation products during O2 tests.

Conclusions & outlook

Progress has been made in quantifying the oxidation of 3D carbon weaves being developed for new generation NASA TPS. Experimental observations and microscale analyses showed a significant decomposition of the weaves by mechanical removal of oxidized fibers in weakened tows. Future efforts will be focused at expanding the study to the oxidation and nitridation of carbon weaves under the effect of O- and N-atoms.

References

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